Zachariasen (1963) refined in the latter stages to 5.646 (1) × 10^{-6}. A final difference Fourier map showed maximum excursions of ± 0.340 e Å^{-3}. Neutral atom scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV), for H atoms from Stewart, Davidson & Simpson (1965), were used as incorporated in a locally modified version of the Enraf–Nonius *SDP-Plus* (Frenz, 1987) which was the source of all programs. A refinement using all 2222 observations, not marked as weak in a prescan, led to $R = 0.082$, $wR = 0.091$, $S = 1.571$. Atomic coordinates are presented in Table 1,* bond distances and angles are listed in Table 2. A view of the molecule is provided in Fig. 1.

Related literature. Sesamin was first isolated from sesame oil. The structure and properties of sesamin have been the subject of a review article (Budowski, 1964). The structure of the related (—)-dihydrosesamin has been determined crystallographically (Lin-Gen, Seligmann, Lotter & Wagner, 1983). The molecule is known to enhance the effects of pyrethroid insecticides.

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* Lists of structure factors, anisotropic displacement parameters, bond distances and angles involving H atoms, and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54632 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0541]

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Structure of 4-Methoxybenzophenone

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Abstract. $C_{14}H_{12}O_2$, $M_r = 212.25$, triclinic, $P\bar{1}$, $a = 9.636$ (5), $b = 13.906$ (4), $c = 9.293$ (4) Å, $\alpha = 108.25$ (2), $\beta = 109.91$ (2), $\gamma = 88.17$ (3)°, $V = 1108.0$ (8) Å^3, $Z = 4$, $D_x = 1.27$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.79$ cm^{-1}, $F(000) = 448$, $T = 296$ K, $R = 0.046$ for 2504 observed unique reflections with $I > 3\sigma(I)$. In one of the two crystallographically independent 4-methoxybenzophenone (MOBP) molecules the plane of the carbonyl group makes dihedral angles of 21.0 and 34.2° with the *p*-methoxyphenyl and phenyl ring planes; corresponding angles in the other MOBP molecule are slightly different: −38.5 and 24.9°.

Experimental. A colorless transparent crystal, with dimensions 1.5 × 0.7 × 0.5 mm, was grown from alcohol solution and mounted in a random orientation on a glass fiber. Data were collected with a Rigaku AFC5R diffractometer (*CONTROL* software; Molecular Structure Corporation, 1988). Cell constants were obtained by least-squares analysis of 20 diffraction maxima ($3 \leq 2\theta \leq 50°$). $\omega/2\theta$ scans, scan width $(1.260 + 0.35\tan\theta)°$, $2\theta \leq 50°$ ($0 \leq h \leq 11$, $-16 \leq k \leq 16$, $-10 \leq l \leq 10$). The intensities of 3915 unique reflections were measured. Three standard reflections were measured periodically; corrections were applied to the intensities to allow for the drop of 1.0% in the mean standard intensities during data collection. An empirical absorption correction, based on azimuthal scans of three reflections, was applied (range of transmission factors from 0.9152 to 1.0000). The data were corrected for Lorentz and polarization factors. 2504 reflections with $I \geq 3\sigma(I)$ were obtained and used in the refinement.

The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983), the C and O atoms being located in the *E* map. H atoms were placed in geometrically calculated positions with C—H = 0.95 Å, but were not included in the refinement. The structure was refined on *F* by the full-matrix least-squares technique with anisotropic thermal parameters for C, N and O atoms. Final $R = 0.046$, $wR = 0.055$ and $S = 1.35$, $w = 1/\sigma^2(F_o)$. $(\Delta/\sigma)_{\max} = 0.006$, 289 parameters; in the final difference synthesis the

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largest and smallest heights were 0.25 and $-0.35\ \mathrm{e\ Å^{-3}}$. Anomalous-dispersion corrections were not applied. All calculations were performed on a VAX-11/785 computer using the *TEXSAN* (Molecular Structure Corporation, 1987) program package, the scattering factors were taken from Cromer & Waber (1974). The views of the molecule and unit cell were produced by the *ORTEP*II program (Johnson, 1976), as shown in Figs. 1 and 2, respectively. The atomic parameters are listed in Table 1;* important bond lengths and bond angles are given in Table 2.

Related literature. Both 4-aminobenzophenone (ABP) (Frazier & Cockerham, 1987; Su, Pan, Li, He & Huang, 1991) and 4-amino-4-methylbenzophenone (AMBP) (Su, Guo, Pan & He, 1992) crystallize in noncentrosymmetric space groups and have large second-harmonic-generation (SHG) efficiencies, whereas crystals of MOBP belong to a centrosymmetric space group and therefore do not exhibit SHG properties. These differences may reflect the presence of intermolecular hydrogen bonds in crystals of ABP and AMBP but not in those of MOBP.

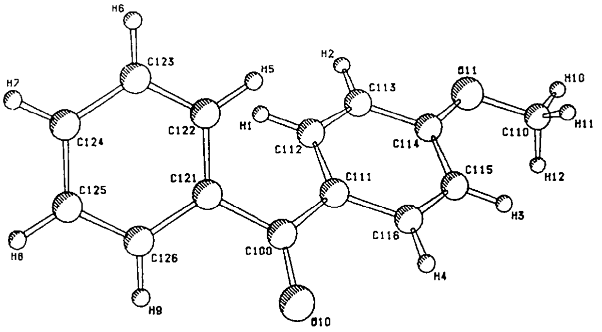

Fig. 1. A view of one of the two independent MOBP molecules. The numbering scheme for the other molecule is derived from that shown here by changing the first digit of the atom number from 1 to 2.

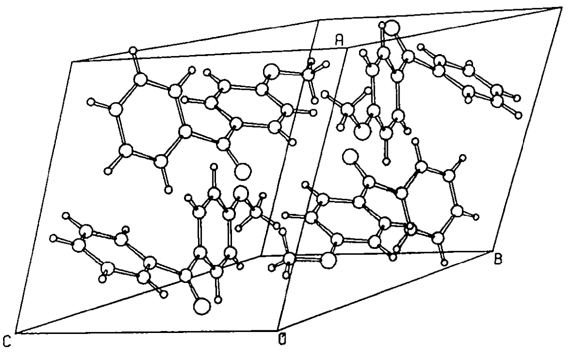

Fig. 2. Packing of MOBP molecules in the unit cell.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2(U_{11} + U_{22} + U_{33}).$$

	x	*y*	*z*	B_{eq}
O(10)	0.5349 (2)	0.2188 (1)	0.4655 (2)	7.06 (8)
O(11)	0.8409 (2)	0.6636 (1)	0.8488 (2)	4.62 (6)
C(100)	0.6365 (2)	0.2495 (2)	0.5949 (3)	4.30 (9)
C(110)	0.8134 (3)	0.7256 (2)	0.7472 (3)	5.8 (1)
C(111)	0.6921 (2)	0.3583 (2)	0.6629 (2)	3.56 (8)
C(112)	0.7691 (2)	0.4068 (2)	0.8274 (3)	3.77 (8)
C(113)	0.8142 (2)	0.5084 (2)	0.8855 (2)	3.75 (8)
C(114)	0.7869 (2)	0.5648 (2)	0.7806 (2)	3.47 (8)
C(115)	0.7097 (2)	0.5183 (2)	0.6167 (2)	3.84 (8)
C(116)	0.6633 (2)	0.4168 (2)	0.5605 (2)	3.82 (8)
C(121)	0.7020 (2)	0.1773 (2)	0.6843 (3)	3.79 (8)
C(122)	0.8531 (2)	0.1800 (2)	0.7631 (3)	4.03 (8)
C(123)	0.9115 (3)	0.1064 (2)	0.8319 (3)	4.7 (1)
C(124)	0.8192 (3)	0.0306 (2)	0.8255 (3)	5.5 (1)
C(125)	0.6688 (3)	0.0274 (2)	0.7488 (3)	5.5 (1)
C(126)	0.6102 (3)	0.0996 (2)	0.6770 (3)	4.7 (1)
O(20)	0.0237 (2)	0.2209 (1)	0.5018 (2)	5.92 (7)
O(21)	0.3527 (2)	0.6601 (1)	0.8501 (2)	5.03 (6)
C(200)	0.1362 (2)	0.2489 (2)	0.6207 (3)	3.97 (8)
C(210)	0.2547 (3)	0.7379 (2)	0.8366 (4)	6.6 (1)
C(211)	0.1950 (2)	0.3577 (2)	0.6845 (2)	3.42 (7)
C(212)	0.3459 (2)	0.3887 (2)	0.7547 (3)	3.57 (8)
C(213)	0.3942 (2)	0.4896 (2)	0.8054 (2)	3.68 (8)
C(214)	0.2927 (2)	0.5619 (2)	0.7913 (2)	3.64 (8)
C(215)	0.1434 (2)	0.5327 (2)	0.7216 (3)	3.88 (8)
C(216)	0.0955 (2)	0.4309 (2)	0.6666 (3)	3.85 (8)
C(221)	0.2099 (2)	0.1754 (2)	0.7032 (3)	3.71 (8)
C(222)	0.1838 (3)	0.0721 (2)	0.6161 (3)	4.7 (1)
C(223)	0.2391 (3)	0.0012 (2)	0.6919 (4)	5.7 (1)
C(224)	0.3205 (3)	0.0317 (2)	0.8545 (4)	5.4 (1)
C(225)	0.3472 (3)	0.1336 (2)	0.9424 (3)	4.7 (1)
C(226)	0.2932 (2)	0.2055 (2)	0.8673 (3)	3.98 (8)

Table 2. *Intramolecular distances* (Å) *and angles* (°)

O(10)—C(100)	1.221 (3)	O(20)—C(200)	1.220 (3)
O(11)—C(110)	1.424 (3)	O(21)—C(210)	1.420 (3)
O(11)—C(114)	1.355 (3)	O(21)—C(214)	1.366 (3)
C(100)—C(111)	1.483 (3)	C(200)—C(221)	1.487 (3)
C(100)—C(121)	1.491 (3)	C(200)—C(211)	1.492 (3)
C(111)—C(116)	1.391 (3)	C(211)—C(216)	1.386 (3)
C(111)—C(112)	1.398 (3)	C(211)—C(212)	1.394 (3)
C(112)—C(113)	1.370 (3)	C(212)—C(213)	1.373 (3)
C(113)—C(114)	1.388 (3)	C(213)—C(214)	1.384 (3)
C(114)—C(115)	1.390 (3)	C(214)—C(215)	1.376 (3)
C(115)—C(116)	1.373 (3)	C(215)—C(216)	1.381 (3)
C(121)—C(122)	1.385 (3)	C(221)—C(226)	1.390 (3)
C(121)—C(126)	1.388 (3)	C(221)—C(222)	1.392 (3)
C(122)—C(123)	1.380 (3)	C(222)—C(223)	1.377 (4)
C(123)—C(124)	1.375 (3)	C(223)—C(224)	1.375 (4)
C(124)—C(125)	1.375 (4)	C(224)—C(225)	1.377 (3)
C(125)—C(126)	1.377 (3)	C(225)—C(226)	1.384 (3)

C(114)—O(11)—C(110)	118.2 (2)	C(125)—C(126)—C(121)	120.3 (2)
C(214)—O(21)—C(210)	118.0 (2)	O(20)—C(200)—C(221)	119.8 (2)
O(10)—C(100)—C(111)	119.8 (2)	O(20)—C(200)—C(211)	119.3 (2)
O(10)—C(100)—C(121)	119.5 (2)	C(221)—C(200)—C(211)	120.9 (2)
C(111)—C(100)—C(121)	120.6 (2)	C(216)—C(211)—C(212)	118.4 (2)
C(116)—C(111)—C(112)	117.6 (2)	C(216)—C(211)—C(200)	118.5 (2)
C(116)—C(111)—C(100)	119.1 (2)	C(212)—C(211)—C(200)	123.0 (2)
C(112)—C(111)—C(100)	123.2 (2)	C(213)—C(212)—C(211)	120.6 (2)
C(113)—C(112)—C(111)	121.1 (2)	O(22)—C(213)—C(214)	120.0 (2)
C(112)—C(113)—C(114)	120.2 (2)	O(21)—C(214)—C(215)	124.6 (2)
O(11)—C(114)—C(113)	115.8 (2)	C(21)—C(214)—C(213)	115.2 (2)
O(11)—C(114)—C(115)	124.6 (2)	C(215)—C(214)—C(213)	120.2 (2)
C(113)—C(114)—C(115)	119.6 (2)	C(214)—C(215)—C(216)	119.6 (2)
C(116)—C(115)—C(114)	119.5 (2)	C(215)—C(216)—C(211)	121.1 (2)
C(115)—C(116)—C(111)	121.9 (2)	C(226)—C(221)—C(222)	118.9 (2)
C(122)—C(121)—C(126)	118.9 (2)	C(226)—C(221)—C(200)	122.2 (2)
C(122)—C(121)—C(100)	121.9 (2)	C(222)—C(221)—C(200)	118.6 (2)
C(126)—C(121)—C(100)	119.0 (2)	C(223)—C(222)—C(221)	120.4 (2)
C(123)—C(122)—C(121)	120.5 (2)	C(224)—C(223)—C(222)	120.4 (2)
C(124)—C(123)—C(122)	120.0 (2)	C(223)—C(224)—C(225)	120.0 (2)
C(123)—C(124)—C(125)	120.1 (2)	C(224)—C(225)—C(226)	120.1 (2)
C(124)—C(125)—C(126)	120.2 (2)	C(225)—C(226)—C(221)	120.2 (2)

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54576 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Methyl-9-phenyl-1,3,8,9-tetraazabicyclo[5.3.0]dec-7-en-10-one

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Abstract. $C_{13}H_{16}N_4O$, $M_r = 244.30$, orthorhombic, $P2_12_12_1$, $a = 8.6562\ (3)$, $b = 11.0875\ (4)$, $c = 12.8259\ (10)$ Å, $V = 1231.0\ (1)$ Å^3, $Z = 4$, $D_x = 1.308$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0.71069$ Å, $\mu = 0.82$ cm^{-1}, $F(000) = 520$, $T = 293$ K, $R = 0.036$ for 1452 observed reflections. The molecule consists of a five- and a six-membered ring, which are planar [maximum deviations 0.005 (2) and 0.007 (2) Å, respectively], and a seven-membered ring, which adopts a chair-like conformation. The N(3)═C(8) and O═C(7) double bonds are clearly defined by bond lengths [1.296 (3) and 1.222 (3) Å, respectively].

Experimental. The title compound belongs to the class of 4,5-heteroannulated 2,3-dihydro-2-phenyl-4*H*-1,2,4-triazol-3-ones. The compound has been obtained as the sole product by potassium permanganate oxidation of 7-methyl-2-phenyl-1,2,4,7-tetraazaspiro[4.5]decan-3-one (Seil, 1989; Gstach & Seil, 1990*a*). The crystals were grown from propan-2-ol. Pale yellow crystal, 0.2 × 0.3 × 0.5 mm. Cell dimensions from setting angles of 32 reflections having $10 < 2\theta < 30°$. Data collection on a Phillips PW 1100 four-circle diffractometer, Mo $K\alpha$ radiation, graphite monochromator, ω–2θ scans, scan width 1.2°, scan speed 2° min^{-1}, $4 < 2\theta < 48°$, $0 \le h \le 9$, $-12 \le k \le 12$, $-14 \le l \le 14$, 4135 reflections, 1937 independent reflections ($R_{\text{int}} = 0.021$), 1452 with $I > 3\sigma(I)$ observed. The intensities of three standard reflections (112, 222, 213) measured every 2 h were found to be constant within 2%. Data corrected for Lorentz and polarization effects, but not for absorption.

The structure was solved by direct methods and difference Fourier syntheses and refined by least-squares methods, based upon F with weights $w = [\sigma^2(F_o) + 0.00033F_o^2]^{-1}$ (Sheldrick, 1976). H atoms included using a riding model (C—H = 1.08 Å, H—C—H = 109.5°, phenyl H atoms on the external bisector of the appropriate C—C—C angle). C, N and O atoms anisotropic, H atoms isotropic. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0.036$, $wR = 0.035$, maximum and minimum electron density in final difference Fourier map 0.13 and -0.12 e Å^{-3}, respectively. $(\Delta/\sigma)_{\max} = 0.005$.

Final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Bond

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[*] Lists of structure factors, anisotropic thermal parameters and positional and isotropic thermal parameters for the H atoms, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54484 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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